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A study of process and techniques of transition metal oxides

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Abstract
Nano structured transition-metal oxides display magnificent properties, for example, ferromagnetic, ferroelectric, photoluminescence and semi conductive practices, and so on. The band hole and electronic structure of these oxides can be controlled by size and measurements; they can likewise be utilized as a part of an extensive variety of utilizations including microelectronics, energy storage, sensors, and biomedicine because of their tunable concoction and physical properties. Here, we give a far reaching survey of the controllable growth of some common change metal oxide nanostructures. At that point impact of oxygen plasma treatment on the mechanical properties of ZnO nanorods has been examined. A ZnO nanorods was used to enhance the yield vitality. Co3O4 nanostructures though the impact of anions and used one of the nanostructure to build up a quick and solid pH sensor. At last to take the benefit of higher level of redox science of NiCo2O4 contrasted with the single period of nickel oxide and cobalt oxide, a delicate glucose sensor is created by immobilizing glucose oxidase. Be that as it may, there were issues with the mechanical vigor, lifetime, yield steadiness and environmental versatility of such gadgets, in this way more work is going ahead to discover new ways and means keeping in mind the end goal to enhance the execution of created Nano generators and sensors.

Keywords: techniques, metal oxides, nanostructure, biomedicine, applications, energy storage

Introduction

The characterization of as grow nanostructures for inside and out learning with respect to their morphology and different properties has been to a great extent in light of number of methods and techniques that were developed for this purpose. These incorporate X-beams diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), atomic for microscopy (AFM), X-ray photo electron spectroscopy (XPS), cathode glow (CL) and Nano indentation. These featured methods are only those that were utilized as a part of the exhibited work. Following is a short talk on every one of them with respect to their utilization and acquired data.

In the course of recent decades, change metal oxide nanostructures (MONSs), which have extraordinary potential in magnetic, electronic, and optical applications, have been generally contemplated. MONSs have been coordinated into an assortment of gadgets to accomplish uncommon great execution, for example, improved gas detecting and proficient photo catalysis. Experiencing significant change metal oxides, in spite of the fact that the s-shells of positive metallic particles are dependably completely filled by electrons, the d-shells of them might be not totally filled. This trademark brings them different interesting properties, which include responsive electron transitions, high dielectric constants, wide band holes, and great electrical attributes et cetera. In the interim, progress metal oxides possess various states, for example, ferromagnetic state, ferromagnetic state and semi conductive state. Thusly, progress metal oxides are thought to be a standout amongst the most interesting functional materials.

Presently, the examinations nanostructures have enormously advanced the improvement of electron gadgets. Nanostructures are characterized as the materials with no less than one measurement between and 100 nm. All in all, nanostructures possess three unique morphologies: zero-dimensional (0D), one-dimensional (1D), and two-dimensional (2D) nanostructures. It has been notable that with the size and measurement decreased, the electronic structures of the nanostructures can be tuned, which prompt an assortment of changes in both concoction and physical properties. For instance, magnetic properties of MONSs can change from Ferro/ferromagnetic to super paramagnetic with their size decreased. Along these lines the Nano scale plan and procedure of blend can be utilized to tune the properties of nanostructures.

Review of literature

X-ray diffraction (XRD): Typically X-ray is an electromagnetic radiation having a wave length of 1å in the middle of bright and gamma-beams. In material science X-beams diffraction is known as a portrayal procedure fit for examining the crystalline structures of the developed nanostructures. This non-ruinous analytical technique is very helpful for concentrate concoction organization, precious stone structures and their stages, measure, symmetry of the unit cell, cross section constants of nanoparticles and physical properties of developed materials. It is critical to bring up that over 90% strong materials are crystalline in nature [1] and each crystalline has a remarkable X-beam diffraction design that can be utilized simply like "unique finger impression" with a specific end goal to distinguish the material. The cooperation of X-beam shaft with precious stone brought about a

diffraction design [2] that recognizes the material and comparing stage. The data got from the diffraction design incorporates size of the crystallite, symmetry of the unit cell, stress, strain and development introduction, and so forth. The working marvel behind the age of diffraction example can be depicted along these lines. At the point when a X-beam shaft having wave length?? Hits the strong precious stone with an edge? the came about scattered radiation can be controlled by ideals of Bragg's law (i.e. ???? = 2?? ?????? ??). Where n is known as the diffraction request and d means the separation in the middle of the diffracting planes. Keep in mind that the arrangement of d-planes is novel for every single material. It is essential to call attention to that by controlling diverse parameters like the geometry of episode beams and the introduction of the indicator and precious stone, one can acquire all the conceivable diffraction headings of the grid [2, 3]. In the exhibited work a Phillips PW 1729 powder diffract meter outfitted with Cu Kα radiation (λ=1.5418 Å) having a generator voltage of 40 kV and current of 40 mA has been utilized.

Scanning electron microscope (SEM): Scanning electron microscope (SEM) is a standout amongst the most critical instruments so as to think about the general appearance of the grown nanostructures. It encourages us in investigating diverse parameters like quality, shape, thickness, and distance across, thickness, length and introduction of the as grown nanostructures. SEM has a place with the group of microscopes, but it utilizes a light emission in lieu of light keeping in mind the end goal to make a picture. The light emission goes through the electromagnetic focal points and strikes the surface of the example. The assault of electrons does not makes any harm the examples. The identifier gathers the optional/backscattered electron ejected from the example and believers them into a flag. At long last this flag is coordinated towards displaying screen [4]. SEM can catch the pictures in the scope of noticeable to few nanometers, while the amplification go is around 20X-30000X alongside a spatial determination of 50-100nm [5-7]. The electron acceleration voltages are typically in the scope of 5-20 kV. Additionally points of interest can be found in ref [8]. In the exhibited work SEM (show LEO 1550 Gemini magnifying lens) running at 15 kV was utilized to examine the morphology of the as grown nanostructures.

Transmission electron microscope (TEM): Transmission electron microscope (TEM), like SEM, has a place with the group of electron microscopes. With a specific end goal to get basic data at nuclear level TEM is a standout amongst the most ordinarily used techniques. The working standard of TEM is much the same as optical magnifying lens yet with a moderately high energy electron wellspring of around 200 KeV. The TEM is likewise extremely important apparatus for the material's analysis, as one can get assortment of data including chemical composition [9], gem/surface structure information and images up to few angstrom resolutions just by exchanging the operational mode. In correlation with SEM, TEM is multi-reason and has much enhanced amplification and determination. Be that as it may, dissimilar to SEM, TEM has a few detriments too, which must be kept under thought. Maybe a couple of them are highlighted here.

1. It is a destructive technique.
2. It is also time consuming (in order to prepare sample).
3. Only provides local information.
4. Relatively difficult to operate.

In the presented work the measurements were conducted by using TEM (model FEI Tecnai G2 TF20 UT) accompanied with field emission gun running at 200 kV with a point resolution of 1.9Å, and also equipped with energy dispersive spectrum (EDS).

Atomic force microscope (AFM): Atomic force microscope (AFM) is a characterization tool that has been extensively used for imaging, measuring, and manipulating samples at the Nano scale. Some of the notable features of AFM are highlighted here.

1. It is a non-destructive technique.
2. Useful for measuring the surface profile of the material with high resolution three dimensional images.
3. Useful for measuring the force at the Nano-newton level.
4. No particular sample preparation is needed.
5. Measurements can be performed at room temperature.
6. All kinds of substrates like hard and soft, conductive and isolative can be investigated.

The working of AFM is subject to silicon made cantilever with a sharp platinum covered tip at one of its finishes. At the point when this tip is acquired the region of the surface of the example, the cantilever is avoided because of the Van der Waals compel and the laser is in charge of catching the extent of redirection. It is essential to call attention to that the checking stature of the tip isn't steady keeping in mind the end goal to maintain a strategic distance from the crash of tip with the surface of the example. Along these lines there exists an input system keeping in mind the end goal to alter the separation between the tip and the surface of the example, so the power between the tip and the surface of the example stay consistent. There are two working styles of AFM known as contact mode and tapping mode. In contact mode the tip touches the surface of the example, while in tapping mode the tip tapes over the surface of the example. In the exhibited work the piezoelectric estimations were recorded in contact mode by utilizing a Digital Instruments Multimode AFM (Netherlands) and a hand crafted trans-impedance enhancer alongside hardened platinum covered tests (NTMDT NSG11/Pt, Russian Federation).

Nano indentation: Nano indentation method is an altered AFM strategy. In this procedure the cantilever utilized as a part of AFM has been supplanted with a Nano indentation gadget known as hysteron tribo scope. This Nano indentation gadget is pushed on the material so as to get data about the hardness and flexible modulus of the material. That is the reason this technique has been utilized frequently for the portrayal of the mechanical properties of the material [12]. In the displayed work Nano indentation was performed by utilizing a Triboindenter TI-950 (Hysitron) with a conductive boron-doped precious stone Berkovich tip of ~ 3 Ω cm resistivity.

Cathodo luminescence (CL): Cathodo luminescence (CL) is a nondestructive technique used to discover the source of the radiance from a specific piece of the grown nanostructures. CL technique records radiance on the barrage of high energy electron through a cathode firearm keeping in mind the end

goal to make electron– opening sets that reason light outflow. The subsequent glow gives surfaces and compositional varieties, which are unrealistic through light microscopy. CL is likewise exceptionally helpful in getting profound understanding of pollution actuated imperfections and expanded deformities. In the displayed work the CL estimations were performed by utilizing a MonoCL4 framework incorporated with a LEO 1550 Gemini SEM and furnished with a quick CCD recognition framework or a Peltier cooled PMT for otherworldly securing.

Oxygen plasma treatment: The developed ZnO NRs were treated with oxygen plasma through responsive particle drawing (RIE) framework (SAMCO, RIE-10RU). The RIE framework was outfitted with the parallel plate compose plasma reactor chamber. The oxygen plasma was incited for just three minutes by setting alternate parameters as RF energy of 250 W, RF frequencies of 13.56 MHz, a gas stream of 400 sccm and a weight of 600 Pa. The primary goal of treating ZnO NRs with the oxygen plasma was to decrease the imperfections levels in the as developed ZnO NRs and to watch its impact on the piezoelectric properties of ZnO.

Growth process

Substrate treatment: The substrate is the center of the combination procedure. The treatment of the substrate preceding development process has significant effect on the morphology and nature of the developed nanostructures. Some vital advances identified with substrate treatment are featured here.

Cleaning of substrate: The fundamental motivation behind the cleaning is to evacuate/wipe out the inconspicuous tidy or natural contaminant or some other obscure particles (if show there). Cleaning of substrate preceding development has awesome significance for getting high caliber, thick, uniform, surrenders free and all around adjusted nanostructures as the tidy particles and other undesirable chemicals/particles display on the surface of the substrate can harm the quality, dependability, arrangement and consistency of the developed nanostructures [10]. Cleaning likewise assumes a critical part to reproduce the nanostructures. The cleaning has been performed by means of ultrasonic shower by utilizing CH3)2CO and isopropanol separately for 5 minutes every, at that point washed with deionized water and dried by stream of nitrogen gas.

Deposition of conductive layer: Now and again there is a need of conductive layer on the substrate keeping in mind the end goal to use as a base cathode amid the estimations. For instance on the off chance that we are getting ready examples for the creation of Nano generators then we ought to have a conductive substrate [11]. So a metal evaporator (Satis-having a weight of 2.5x10-6 mbar) has been used to make surface of the substrate conductive by setting a layer of any conductive material, for example, Silver, Platinum, Gold, and Aluminum. When the testimony of conductive layer it is obligatory to clean the substrates by rehashing an indistinguishable procedure from clarified previously.

Preparation of seed solution

1. **For ZnO nanorods:** The seed arrangements can be set up by utilizing diverse solvents and forerunners. We used two various types of seed answers for the development of ZnO nanorods. To begin with seed arrangement was set up by dissolving 5 mM of zinc acetic acid derivation dihydrate (Zn (CH3COO) 2.2H2O) in unadulterated ethanol arrangement as revealed by Green *et al.* This seed arrangement was utilized for hard substrates like Glass, Silicon, FTO and ITO; on the grounds that these substrates require pre development strengthening keeping in mind the end goal to deteriorate zinc acetic acid derivation dihydrate into ZnO nanoparticles. Another seed arrangement was set up by following the technique for Pacholski *et al.* For this situation, we broke up 5 mM of zinc acetic acid derivation dihydrate and KOH in unadulterated methanol arrangement. This sort of seed arrangement is reasonable for delicate/adaptable substrates like regular paper, plastic, material fiber and aluminum thwart, since zinc acetic acid derivation dihydrate proselytes to ZnO nanoparticles at room temperature.
2. **For Co3O4 nanostructures:** A seed crystal solution was prepared by dissolving 274 mg of cobalt acetate anhydrous in 125 ml methanol and left for stirring at a temperature of 60°C for two hours. After two hours cobalt acetate anhydrous was dissolved completely and a uniform blue color solution was appeared.
3. **For NiCo2O4 nanostructures:** A seed crystal solution was prepared by dissolving 274 mg of cobalt chloride hexahydrate in 125 ml methanol and left for stirring at a temperature of 60°C for two hours. After two hours cobalt chloride hexahydrate was dissolved completely and a uniform blue color solution was appeared.

Deposition of seed solution: Two drops of the readied seed arrangement were connected on the substrate by utilizing a turn coater (Laurell WS-650-8B) running at around 4000 r.p.m. The procedure was rehashed three times for 30 seconds each time. The thickness/surface scope of the seed layer can be safeguarded by modifying the turning speed. The principle motivation behind utilizing seed layer is to supply nucleation destinations by weakening the thermodynamic obstruction between heterogeneous materials [3]. Another preferred standpoint that has been watched is that when seed layer was utilized, the developed nanostructures were observed to be very much adjusted, exceedingly thick and uniform.

Synthesis of ZnO nanorods: In the wake of directing a progression of investigations following conditions were advanced to get very much adjusted, thick and uniform development of ZnO nanorods. An equi-molar (0.075) arrangement of hexamethylenetetramine (C6H12N4) and zinc nitrate hexahydrate (Zn (NO3)2. 6H2O) were broken down in 100ml deionized water and left to stir until the point that the arrangement ends up straightforward. At that point substrates enhanced with ZnO seed particles were put in a measuring glass confronting descending by the assistance of a Teflon test holder. After that the measuring utencil was kept in a preheated electric broiler at 95°C for 5– 6 hours. After the finishing of the development length the substrates were washed precisely with the deionized water to expel leftover strong particles from the surface. At long last, the examples

were dried in air at room temperature.

Synthesis of Co3O4 nanostructures: The development answers for various cobalt salts have been set up by taking equi-molar (0.1 M) convergence of urea with antecedents including cobalt nitrate, cobalt acetic acid derivation, cobalt chloride and cobalt sulfate, each in 50 ml of deionized water and after that all arrangements were left on mixing for 30 min. The Silicon substrates adorned with Co3O4 particles were set in these development arrangements by the assistance of a Teflon test holder confronting descending. At that point tests were kept in preheated electric broiler at 95°C for 5– 6 hours. After the development term tests with the Co3O4 nanostructures were taken out from the development arrangement and washed in the deionized water keeping in mind the end goal to expel remaining strong particles from the surface. At that point, the examples were dried in air at room temperature.

Synthesis of NiCo2O4 nanostructures: Watery compound development technique has been utilized for the development of NiCo2O4 nanostructures on nickel froth as substrate. The forerunner arrangement was set up by dissolving 2.37 g of cobalt chloride hexahydrate, 1.185 g of nickel chloride hexahydrate and 2.7 g of urea in 75 mL of deionized water and afterward the arrangement was left on mixing for 30 min. From that point onward, the nickel froth pieces finished with seed particles of cobalt chloride hexahydrate were set in the measuring glass containing antecedent arrangement by the assistance of a Teflon test holder confronting descending and the recepticle was kept in a preheated broiler at 95°C for 5– 6 h. After the development time frame nickel froth pieces with the NiCo2O4 nanostructures were taken out from the development arrangement and washed in the deionized water keeping in mind the end goal to expel lingering strong particles from the surface. At last, the examples were dried in air at room temperature.

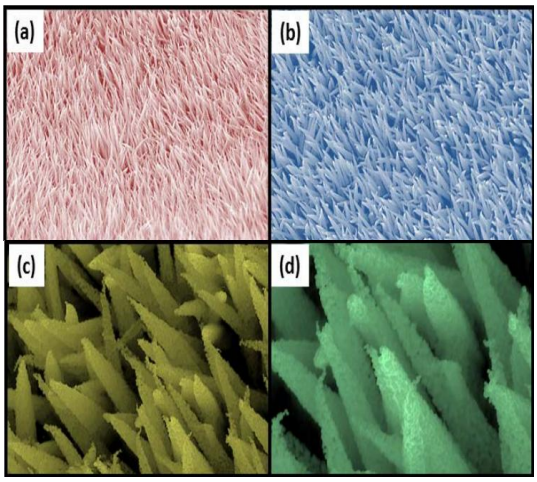

Fig 1: SEM images of $NiCo_2O_4$ nanostructures

Conclusion

The growth of metal oxide nanostructures by using low temperature aqueous chemical growth method. Aqueous chemical growth method is the most simple, cheap and effective method to synthesize different metal oxide nanostructures. ZnO nanostructures are more preferred for energy harvesting applications. Oxygen plasma treatment is an effective way in order to reduce the defects levels in the grown ZnO nanostructures, which ultimately affects its piezoelectric properties and an enhancement in the amount of piezoelectricity has been observed. Beside this the effect of oxygen plasma on mechanical properties has also been investigated. Flexible and cheap substrates like paper, plastic, textile fabric can be successfully utilized in the fabrication of piezoelectric Nano generators. ZnO nanorods grown AFM tip is also a good alternative in order to get an enhanced amount of piezoelectricity. Co3O4 is a potential metal oxide with variety of nanostructures. The features associated with Co3O4 like high surface area, short path length for ion transport and easily tunable surface have made Co3O4 a promising material for electrochemical devices. Therefore in order to get maximum advantage of these properties a pH sensor has been developed.

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